

1045.016



# PATENT SPECIFICATION

DRAWINGS ATTACHED

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## COMPLETE SPECIFICATION

### Improvements in or relating to Mirrors

We, ARTHUR ELLIOTT, a British Subject of, Department of Biophysics, University of London King's College, 26—29, Drury Lane, London, W.C.2, and UNIVERSITY OF LONDON KING'S COLLEGE, a British University College, of Strand, London, W.C.2, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the production from optically worked negative masters of plated resin replicas which are suitable for use as mirrors for reflecting and focusing radiation such as light; infra-red and ultra-violet radiation and X-rays, and also relates to X-ray diffraction cameras including such mirrors.

A method for producing plated resin replica mirrors has been proposed in which a replica of a negative master was cast in several layers of epoxy resin on a stout backing plate, the negative master first having been coated with a release agent to enable the replica to be separated from the master without damage, and in which the replica was coated with a reflecting surface of aluminium after separation from the master. As the surface actually copied in this method was that of the release agent coating the master it was necessary for this coating to be perfectly uniform in thickness if the replicas were to be accurate copies of the master.

According to the principal aspect of the present invention, a replica mirror is formed by coating a cleaned surface of a negative master with a layer of gold, arranging a blank having a surface approximating to that of the master in close non-touching relationship with the master so that points of its surface are in register with corresponding points of the surface of the master, filling the space

between the blank and the negative master with casting resin; curing the resin and then separating the gold coated blank, forming a replica mirror, from the negative master.

We have discovered that the gold coated resin may easily be pulled apart from the negative master, enabling the use of a release agent to be obviated and the gold coating to be deposited directly on the cleaned surface of the negative master. Examples of substances suitable for forming the negative master are glass, fused silica, agate and steel. Sufficiently good reproduction of the surface texture of the negative master can be obtained using a method embodying the present invention for radiation at least as short as 1.5Å to be satisfactorily reflected near grazing incidence. Another advantage of the present invention is that the gold coating adheres firmly to the resin. When gold is applied to a surface by evaporation to make a mirror the adhesion is, in our experience, very poor and subsequent cleaning of the mirror (should it become dirty) is almost impossible.

The gold coating is deposited in a vacuum deposition process and preferably subjected to heat treatment when on the negative master to reduce the possibility of damage when the resin is applied. After heat treatment a very thin coating of resin, like a varnish, is preferably applied to the gold surface for example by dipping and allowed to set before the remainder of the casting is applied by mounting the gold and resin varnished negative master in close proximity to the blank in such a way that a nearly uniform space of the order of a few thousandths of an inch separates the negative master from the blank and filling the space with a suitable commercial casting resin mixed with a hardener.

A process embodying this principal aspect of the present invention allows a number of replica mirrors to be made from one nega-

tive master which is advantageous when the surface of the negative master is an aspheric one and difficult to produce. A further advantage is that a negative master can frequently be more easily manufactured by grinding and polishing than can the positive shape. For example, we have shown that toroidal and ellipsoidal mirrors such as it would be practicable to make by a conventional grinding and polishing process, can be produced by a method embodying the present invention.

An important application of such toroidal or ellipsoidal mirrors is in the field of X-ray diffraction. In the past, exposure times of several hours or even of days have frequently been required in diffraction investigations. The possibility of reducing these times by using cylindrical surfaces for collimating X-ray by reflection at near grazing incidence has been suggested and such mirrors have been used but due to the very low maximum glancing angles the radii of curvature are long and the effective apertures of the mirror are low. However, these apertures may be increased by employing toroidal or ellipsoidal gold coated replica mirrors and methods embodying the present invention make the production of a gold coated mirror for use with a copper K $\alpha$  radiation source practicable, thus enabling the exposure times for high angle diffraction work to be reduced considerably. Toroidal or ellipsoidal mirrors may be used for X-radiation of wave-length greater than that of copper K $\alpha$  radiation, but it is desirable to design a toroid so that the maximum angle of reflection for the chosen wave-length is employed. In this way most of the radiation of shorter wave-length produced by the target will be removed from the beam and a considerable degree of monochromaticity is achieved.

If a higher accuracy of alignment of the optic axis of the reflecting surface of a replica mirror and the longitudinal axis of the cylindrical blank in which it is cast is required than can be achieved by the casting method described, alignment errors may be compensated for in subsequent use by mounting a mirror cast in this way on a leaf spring in a tube and providing screws acting against the natural resilience of the spring or adjusting the precise position of the mirror. The effect of a complete toroid may be obtained by mounting two hemi-toroidal mirrors in a tube in this way to be independently adjustable.

In order that the invention may be more clearly understood a preferred method embodying the invention of making a gold plated replica mirror of toroidal form and a diffraction camera including such a mirror will now be described with reference to the accompanying drawings, in which:

Figure 1 shows a side and end elevations

of a negative master and blank used for forming a mirror of hemi-toroidal form and of such a mirror itself;

Figure 2 is a diagrammatic perspective view illustrating the mounting of the negative master and blank of Figure 1 during the casting of the replica mirror;

Figure 3 is a diagrammatic view of the mirror in which the scale has been exaggerated in the radial direction illustrating the focusing properties of the mirror and associated diaphragms for cutting out the direct beam and for reducing stray radiation;

Figure 4 is a diagrammatic view of a diffraction camera including the mirror and aperture arrangement of Figure 3;

Figure 5 shows an end elevation and a longitudinal section of a hemi-toroid in an adjustable mount; and

Figure 6 shows an end elevation and a longitudinal section of two hemi-toroids in a single mount, with separate adjustments for each.

The negative master 1 of Figure 1 is formed from a glass rod 15 cm long and 3 mm maximum diameter by grinding and polishing so that the central 10 cm of its length has a 20 metre radius of curvature. It has been found to be possible using conventional lapping and polishing techniques to form a master with a good approximation to this ideal shape provided the shape is frequently measured during preparation by means of a sensitive mechanical gauge or by interference methods. After a satisfactory shape has been achieved the master is cleaned carefully with acetone or alcohol and coated with a substantially uniform film of gold in a vacuum deposition process. The gold coated master is then preferably heat treated at 100° C for several hours to reduce the possibility of damaging the coating when the resin backing is applied. After such heat treatment a very thin coating of resin is applied to the gold coating of the master by dipping the master. It is believed that by first applying a thin coating in this way and allowing it to set with its free surface exposed to the atmosphere, the tendency of the gold layer to drag way from the negative master during setting is reduced.

The next stage in the production of the replica mirror is to mount the gold-coated and resin varnished master 1 in close proximity to a blank which in this particular example is a hollow hemi-cylinder 2 of light alloy. A convenient mounting arrangement is illustrated in Figure 2 in which the ends of the negative master 1 are inserted respectively in two supporting cylinders 3 and 4 having the same external radius as the blank 2 and mounted in a Vee-block 5 together with the blank 2. This simple arrangement ensures that the master 1 and the blank 2 are coaxial and the space between them is then filled

with an epoxy casting resin. After the resin has been cured the master is removed by carefully pulling it away from the blank, commencing at one end, leaving the gold coating on the inner surface of the resin to provide a mirror conforming exactly in shape with the external surface of the negative master. Side and end elevations of the mirror are shown in Figure 1, a resin coating 7 having been cast on the internal surface of the blank 2 so that its external surface, which is covered by a thin gold reflecting coat 8, has at every point a maximum radius of curvature of 20 metres and a minimum radius which varies according to the distance of the point from the end of the blank. This surface is toroidal, but approximates to an ellipsoid of revolution.

The focusing effect of a toroidal or hemi-toroidal mirror together with diaphragm and stops included in a diffraction camera is illustrated in Figure 3. A considerable advantage of such a mirror is that it is possible to carry out adjustments with visual radiation, this being impracticable with cylindrical mirror intended for X-ray reflection owing to the small apertures and consequent diffraction effects. In Figure 3 radiation from a source A is limited by a first aperture 10, placed close to the source and having a diameter such that the transmitted beam is just wide enough to fill the toroid aperture, and then reflected at the internal gold reflecting surface 8 of the toroid and brought to a focus at B. A circular disc 11 carried on a tungsten wire of 5 mm diameter is mounted coaxially with the toroid and just large enough to prevent the direct beam of radiation passing through the toroid without being reflected and a guard stop 12 is placed close to the object whose diffraction pattern is to be observed. The stops may, for example, be made of a silver-copper eutectic alloy. The mirror and its associated apertures and stops may be mounted in a vee-block 13 which ensures alignment as shown in Figure 4. In one application the hemi-cylindrical blank 2 in which the mirror with the toroidal reflecting surface is cast has an external diameter of .75 inches and is placed in the block 13 which is of accurately ground cast iron. The first aperture 10, the central stop 11 and the guard stop 12 are also mounted in cylinders of external diameter of .75 inches placed in the vee-block 13 to be coaxial with the mirror. For adjustment, a test cylinder (not shown) of .75 inches diameter and turned down to pointed "centres" at the ends is also mounted in the vee-block so that the points define the axis of the arrangement. A rectangular carbon steel hinge 14 which is deformable by both bending and twisting produced by adjustment of micrometers 15 is provided to rotate the vee-block through small angles about a horizontal axis and a

vertical axis so that the toroid axis can be made to pass through the copper target 16 of the X-ray source. A specimen holder 17 and a film holder 18 are mounted semi-kinematically on two carbon steel cylindrical ground rods which are arranged to be parallel with the groove of the vee-blocks. Adjustment and focusing of the toroid may be done by means of a microscope (not shown) with a 1 inch objective on an extension of which is carried a piece of thallium-activated caesium iodide crystal phosphor. Behind the objective, in the microscope tube a disc of lead glass is permanently mounted. The microscope is provided with an eyepiece graticule having a number of concentric circles of different diameters and is mounted on a bracket which can be clamped kinematically to the camera on three widely-separated feet, so that it can be removed and accurately replaced. The centre of the graticule can be made to coincide with the optic axis by inserting a pointed test-bar as explained above and adjusting the graticule centre (by means of levelling screws on the microscope bracket) to coincide with the image of the pointed centre seen in the microscope. The camera may be enclosed in a chamber which may be evacuated or filled with, for example, hydrogen but it has been found that scatter from air when using copper  $K\alpha$  radiation is so small that this is rarely necessary. This arrangement enables high definition X-ray diffraction patterns produced by  $K\alpha$  radiation from a copper target 16 to be recorded on a film 18 with exposure times of the order of one hour rather than one day. Diffraction patterns of metal foils may be obtained with exposure times as short as five minutes using such an arrangement.

Examples of arrangements for compensating for inaccuracy in the alignment of the reflecting surfaces and the supporting blanks of replica mirrors embodying the present invention are illustrated in Figures 5 and 6 in which fine adjustments for the position of the mirrors within a cylindrical tube 19 are provided. In Figure 5 a blank 2 of diameter say 9/16 inches is mounted to be concave downwards in a tube 19 of external diameter .750 inches with its straight edges resting on the upwardly facing straight edges of a semi-cylindrical tube 20 which is concave upwards and rests on a leaf spring 21 in the base of the tube 19. Four screws 22 pass through the walls of the tube 19 and bear against the outer surface of the blank 2 so that the position of the blank within the tube may be adjusted against the resilience of the leaf spring 21 by adjustment of the screws 22. Preferably the inner ends of the screws 22 which bear on the outer surface of the blank 2 are spherical.

In another arrangement shown in Figure 6, two hemi-toroidal reflecting surfaces are

mounted in one tube 19 effectively to form a complete toroid. Between the facing straight edges of the two blanks 23 and 24 on which the reflecting surfaces are formed, two leaf springs 25 are arranged and the position of each replica mirror within the tube 19 may be adjusted independently against the resilience of these leaf springs 25 by means of four screws. The screws 22 for adjusting the position of the reflecting surface on the upper blank 23 pass through the walls of the tube 19 and act on the outer surface of the blank 23 exactly as described in connection with Figure 5. However, to enable the tube 19 to be mounted in a vee-block to facilitate alignment with other components in a camera, screws 26 for adjusting the position of the mirror on the lower blank 24 are arranged to pass through portions of that blank which extend beyond the gold plated surface and bear against the inner surface of the tube 19. Preferably the screws 26 are headless and access to them is through four holes 27 in the tube 19. Thus each semi-toroid may be independently adjusted so that images reflected by each of them may be made to coincide and the effect of a complete toroid obtained.

The above description relates specifically to the manufacture and application of a length of hemi-toroidal mirror having a semi-circular section but a short length of a toroidal mirror, having a full circular section and with its maximum diameter at one extremity, has been produced satisfactorily by a method embodying the present invention and similar methods could also be used for forming other shaped mirrors such as hemi-ellipsoidal or hemi-paraboloidal mirrors.

The toroidal mirrors here described were intended primarily for use in X-ray diffraction cameras as described but their application will not be limited to this field. For example, they may be used for irradiating small areas of tissue culture in the study of damage caused by X-rays or in connection with X-ray fluorescence micro-analysis where a small sample is irradiated by, for example, 1.54Å radiation and the resulting fluorescent radiation from elements lighter than copper is examined by a counter. Though the above description has referred to copper K $\alpha$  radiation, mirrors made by methods embodying the present invention may be used for reflecting and focusing different wave-lengths from different X-ray sources. They are also suitable for use in infra-red spectrometers.

#### WHAT WE CLAIM IS:—

1. A method for producing plated resin replica mirrors comprising: coating a cleaned surface of a negative master with a layer of gold; arranging a blank having a surface approximating to that of the master in close non-touching relationship with the master so

that points of its surface are in register with corresponding points of the surface of the master, filling the space between the blank and the negative master with casting resin; curing the resin and then separating the gold coated blank, forming a replica mirror, from the negative master.

2. A method according to Claim 1, in which the gold coated negative master is subjected to heat treatment before application of the resin coating.

3. A method according to Claim 1 or Claim 2, in which a thin, varnish-like coating of resin is applied to the gold coated surface of the negative master and allowed to set with its free surface exposed to the atmosphere before the master is arranged in close non-touching relationship with the blank.

4. A method according to any one of the preceding claims, in which the blank has a substantially cylindrical external surface and is arranged in close non-touching relationship with the coated negative master by mounting coaxial extensions from both ends of the coated master coaxially in cylinders each having an external radius equal to the external radius of the cylindrical blank and placing the blank and the master-supporting cylinders in the groove of a vee block so that the coated master is coaxially in register with the blank.

5. A gold plated resin replica mirror produced by a method according to any one of the preceding claims.

6. An X-ray diffraction camera including a replica mirror of toroidal, or ellipsoidal-shaped surface according to Claim 5, arranged coaxially with a source emitting radiation for directing a beam of radiation from the source on a coaxially arranged object and focusing the diffracted radiation on a radiation-sensitive film.

7. An X-ray diffraction camera according to Claim 6, in which the replica mirror is mounted on a deformable member provided in an open ended tube arranged coaxially with the source emitting radiation and means are provided for deforming the said member to adjust the position of the replica mirror within the tube.

8. An X-ray diffraction camera according to Claim 7, including a pair of replica mirrors having hemi-toroidal, or hemi-ellipsoidal shaped surfaces arranged within the open ended tube effectively to form respectively a full toroidal or ellipsoidal reflecting surface, each reflecting surface being arranged to be concave towards the other reflecting surface and the straight edges of the hemi-cylindrical blanks on which the reflecting surfaces are formed being separated by deformable members, and further including means for adjusting the position of each reflecting surface within the open ended tube independently

by adjusting the deformation of the deformable members.

- 5 9. A camera according to Claim 7 or 8, in which means for adjusting the position of at least one replica mirror within the open ended tube comprises screws having spherical ends bearing against the outer surface of the blank upon which the said replica mirror is formed.
- 10 10. A camera according to Claim 8, in which the means for adjusting the position within the open ended tube of at least one replica mirror comprises screws passing through portions of the blank on which the replica mirror to be adjusted is formed outside the reflecting area and having spherical ends bearing against the inner surface of the open ended tube.
- 15 11. A method of making a gold plated replica mirror substantially as herein described
- 20

with reference to Figures 1 and 2 of the accompanying drawings.

12. A gold plated replica mirror substantially as herein described with reference to Figures 1 and 3 of the accompanying drawings. 25

13. An X-ray diffraction camera including a gold plated replica mirror and substantially as herein described with reference to Figures 3 and 4 of the accompanying drawings. 30

14. An X-ray diffraction camera including a gold plated replica mirror substantially as herein described with reference to Figures 3 and 4 together with either Figure 5 or Figure 6 of the accompanying drawings. 35

For the Applicants:

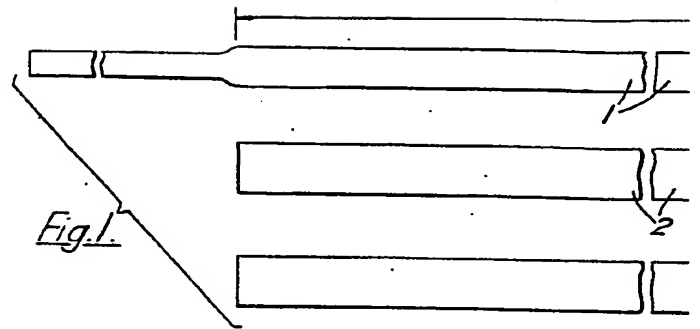
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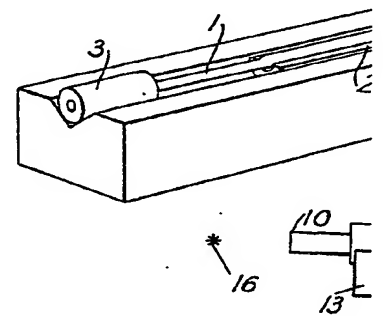
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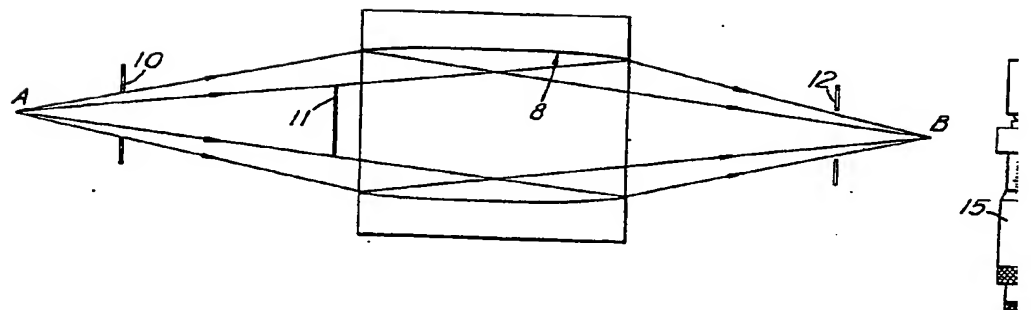
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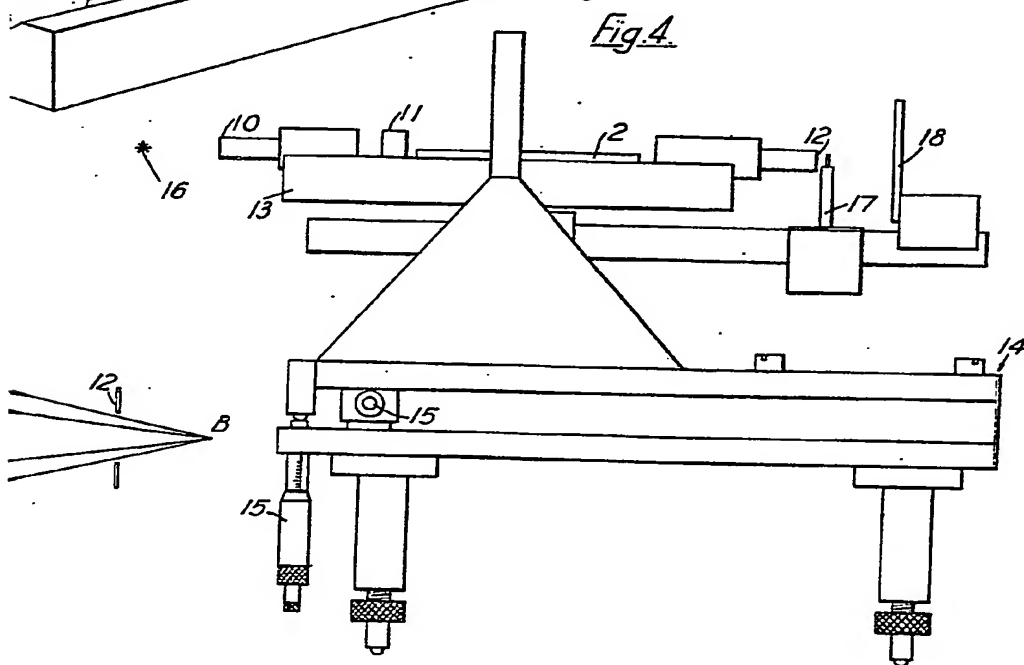
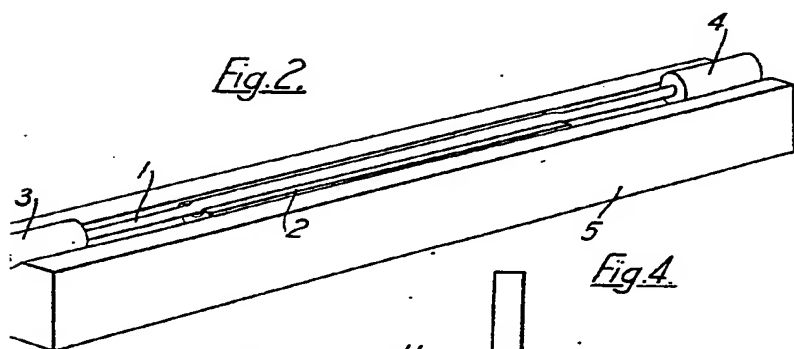
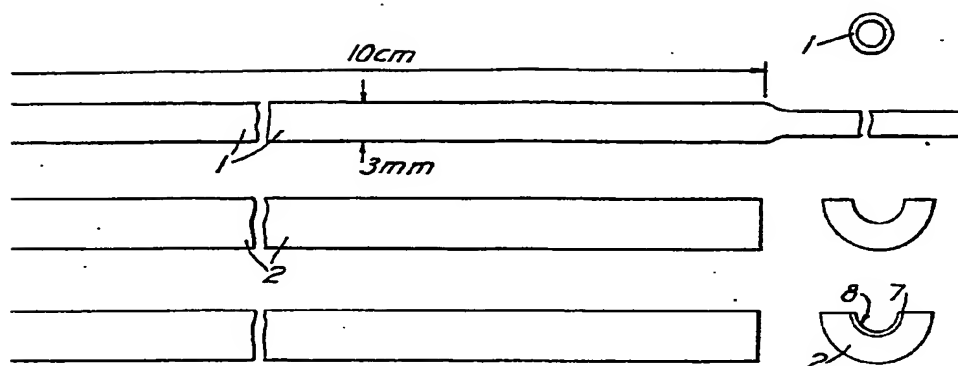


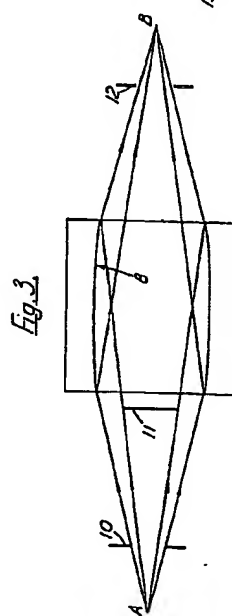
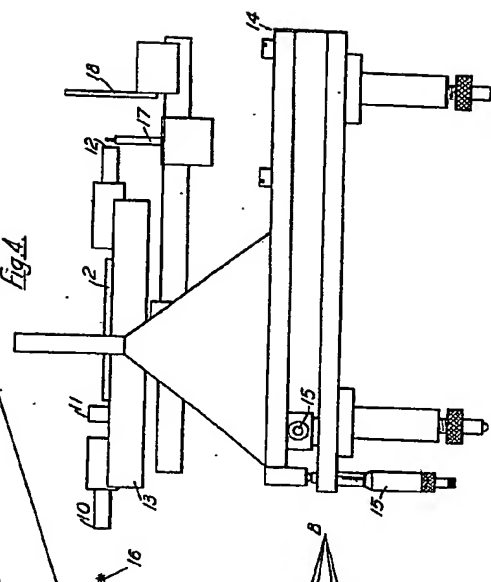
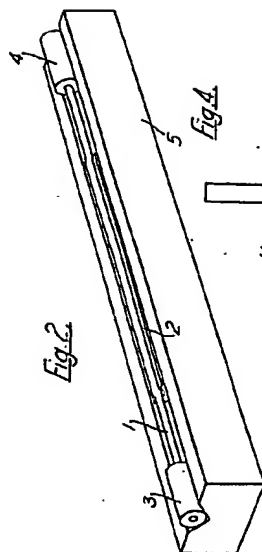
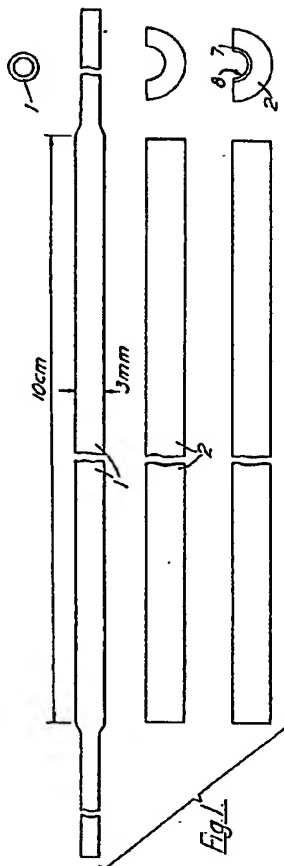
*Fig. 2.*



*Fig. 3.*









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2 SHEETS

COMPLETE SPECIFICATION  
This drawing is a reproduction of  
the Original on a reduced scale  
Sheet 2

